

DATE:

9 OCT 1980

SUBJECT: Annual System Evaluation of the Division of Water Quality, Minnesota Pollution Control Agency and the Division's Contract Laboratory, the Section of Analytical Services, Minnesota Department of Health

FROM: *W. Sanders*
William H. Sanders III, Director
Surveillance and Analysis Division

TO: Charles Sutfin, Director
Water Division

ATTN: Project Officer, Minnesota 106 Grant



On August 19 and 20, 1980, the annual system evaluation was conducted at the Section of Analytical Services, Minnesota Department of Health, Minnesota Pollution Control Agency.

The Quality Assurance Office, Region V, has the responsibility for managing the system evaluation program for the Region. A system evaluation is an on-site inspection and review of the quality assurance program used for the total measurement system to assure that each specific monitoring program conducted under Public Law 92-500, as amended, will produce and document accurate and valid data.

Primary contacts during the on-site system evaluation at the Minnesota Department of Health the Minnesota Pollution Control Agency were:

CONTACT

SPECIALITY

Allen Tupy

Acting Director
Section of Analytical Services
Minnesota Department of Health

Keith Peacock

Unit Leader, Bacteriology
Section of Analytical Services
Minnesota Department of Health

Bill Scruton

Acting Unit Leader
Organic Chemistry
Section of Analytical Services
Minnesota Department of Health

John Davenport

Quality Assurance Coordinator
Minnesota Pollution Control Agency

Barbara Thorsen

Quality Assurance Coordinator
Section of Analytical Services
Minnesota Department of Health

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Dr. David Giese	Assistant Director Division of Environmental Health
Dr. Roger DeRoos	Director Division of Environmental Health
Jean Kahilainen	Senior Chemist Section of Analytical Services Minnesota Department of Health

Region V participants were:

James H. Adams, Chief
Quality Assurance Office
Surveillance and Analysis Division, Region V

Dr. Jane Wells, Statistician
Quality Assurance Office
Surveillance and Analysis Division, Region V

Dr. Emilio Sturino, Chief
Organic Lab Section
Central Regional Laboratory
Surveillance and Analysis Division, Region V

David Payne, Chemist
Quality Assurance Office
Surveillance and Analysis Division, Region V

Maxine C. Long, Microbiologist
Quality Assurance Office
Surveillance and Analysis Division, Region V

The evaluation covered eight major functional areas. These are as follows:

1. Organization
2. Personnel
3. Laboratory Facilities
4. Laboratory Equipment and Instrumentation
5. Methodology
6. Sample Collection, Holding Times, and Preservation
7. Quality Control
8. Data Handling

The FY '79 evaluation report documents in detail, these major functional areas. For a complete review the reader is referred back to this report (dated March 10, 1980). Results of this evaluation will be briefly described. Status of FY '79 deficiencies will be described.

Any new deficiencies will be identified with recommendations for correction. Deficiencies are summarized by function and are to be considered present at the time of the on-site evaluation and not necessarily present at this time. The Quality Assurance Office wishes to emphasize that because of its nature, this report highlights areas of non-compliance rather than the many excellent things that were observed during the evaluation. The staff contacted at the Minnesota Department of Health the Minnesota Pollution Control Agency were very cooperative and helpful throughout the on-site evaluation.

Please address all questions or concerns to James H. Adams, Jr., Chief, Quality Assurance Office at (312) 353-9317.

cc: B. Schade, MPCA
J. Davenport, MPCA
A. Tupy, MN DH
R. DeRoos, MN DH
B. Thorsen, MN DH

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I. Organization

Management responsibilities have been defined. Major monitoring responsibilities are located in the Surface and Groundwaters Section of the Division of Water Quality. The Minnesota Pollution Control Agency contracts with the Minnesota Department of Health for laboratory support.

The Minnesota Pollution Control Agency has had a Quality Assurance Coordinator for some time. The Minnesota Department of Health has recently identified a Quality Assurance Coordinator for the section of Analytical Services. Communications should improve between the laboratory and the Division of Water Quality with each now having in place a Quality Assurance Coordinator. For example the Division of Water Quality Monitoring Staff needs to communicate with laboratory staff before starting special non-routine types of sample collection for special laboratory requirements. Often times no one knows who should be contacted. With the Quality Assurance (QA) Coordinators in place, they should serve as the technical focal point between the two groups.

The laboratory is yet to complete its part of the documentation for the State's total 106 QA program. A draft has been completed of the proposed laboratory QA management document. Methodology documentation is the largest task to be completed.

A very serious problem in the availability of analytical capability to the Pollution Control Agency (or the amount of analytical capability the Pollution Control Agency is willing to pay for) from the Department of Health is noted.

Specifics are discussed in Appendix A, B, and C. This lack of available analytical capability will prevent the Pollution Control Agency from meeting all of their FY '81 monitoring commitments that require laboratory support. The laboratory is working under a personnel handicap. First and foremost is the urgent need to fill the Director's position. Second, the upper levels of management of the Pollution Control Agency and the Department of Health meet and devise an innovative plan that will assure the availability of needed analytical capability so FY '81 monitoring commitments can be met.

- a. Old Deficiency - The Minnesota Pollution Control Agency has not collated a total quality assurance document with quality assurance management responsibility identified for the 106 monitoring effort.

Recommendation - The Minnesota Pollution Control Agency has to impress upon the Minnesota Department of Health the urgent need of finalizing their methodology and QA management documents for collating by the Pollution Control Agency into the total QA program for the complete 106 monitoring effort.

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The task should be completed by December 30, 1980. Document should be formally transmitted to the QAO for review and making recommendations to the Regional Administrator for approval.

II. Personnel

III. Laboratory Facilities

IV. Laboratory Equipment and Instrumentation

V. Analytical Methodology

VI. Quality Control

The above five functional areas are directly related to the laboratory operations. The Minnesota Pollution Control Agency contracts with the Minnesota Department of Health Laboratory, Section of Analytical Services for laboratory support. The evaluation of the Section of Analytical Services is depicted in Appendix A, B, and C.

VII. Sample Collection, Holding Times, and Preservation

Documented field sampling procedures are available for sampling personnel.

a. Deficiency - None

VIII. Data Handling

a. Deficiency - None

In summary, the system evaluation shows progress has been made in correcting deficiencies in the Minnesota Pollution Control Agency contract laboratory and related quality assurance activities of the Division of Water Quality. These deficiencies have an adverse impact on the 106 monitoring activity within the State of Minnesota.

Correction of the identified deficiencies by implementation of the recommendations listed in this report will insure the Minnesota Pollution Control Agency's 106 monitoring activity being in compliance with Agency requirements and producing data that are sufficiently accurate and precise to meet Agency needs.

Please address all questions or concerns to James H. Adams, Jr., Chief, Quality Assurance Office at (312) 353-9317.

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Appendix A

Organic Chemistry Section of Analytical Services Minnesota Department of Health

I. Introduction

Support for the analysis of environmental samples for organic pollutants is provided to the Minnesota Pollution Control Agency by the Organic Unit of the Minnesota Department of Health Laboratory. An on-site evaluation of the unit was performed during August 19 and 20, 1980.

During the on-site evaluation, Mr. Bill Scruton, the Acting Unit Leader indicated that the Unit had the following workload:

- approximately 40 water samples/month for PNA analysis
- two to three samples/month for the Safe Drinking Water Act
- 165 fish samples for PCBs and pesticides
- 65 fish samples for PCBs
- 40 hazardous waste samples for selected organics
- 60 to 80 sediment samples for PCBs and pesticides
- 60 to 80 oil samples for PCBs
- 60 to 80 sediment samples for PCBs and pesticides
- 60 to 80 water samples for PCBs and pesticides

With the workload in consideration, listed below are the observations, conclusions, and recommendations resulting from the on-site evaluation.

III. Personnel

At the time of the on-site evaluation, the Unit was staffed with four permanent and two temporary positions. These positions were filled by the following individuals:

1. Bill Scruton - Acting Unit Leader
2. Vern Terman - responsible for the herbicide analysis

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3. Donna Oman - responsible for pesticide and PCB analysis
4. Sue Skorich - responsible for the polynuclear aromatic hydrocarbon (PNA) instrumental analysis
5. Rich Mohw - responsible for sample preparation
6. David Mehrheim - responsible for sample preparation

Although some of the above personnel could have used more training in the instrumental aspects of the analysis, all were qualified in terms of necessary background (experience) and education to carry out the assigned tasks. However, the number of people assigned to this Unit is not adequate to meet the workload. As a result, some samples (such as the fish) are carried from year to year.

- a. Deficiency - The number of personnel assigned to the Unit is not sufficient to meet the workload on a timely basis.

Recommendation - Assign more personnel to the Unit or decrease the workload.

3. Facilities

The necessary physical facilities are available and adequate to provide a good working environment. The Unit could use more hood space to accommodate the extraction process. This can be accomplished by better utilization of the present hoods. The specific details of the physical facilities are covered in the FY '79 evaluation report (March 10, 1980) of this laboratory and should be consulted if needed.

- a. Deficiencies - None

4. Instrumentation

All of the instrumentation necessary to carry out a detailed organic monitoring program are available in this laboratory. A list of the major instruments is available in last years evaluation report.

The Unit has a Finnigan Model 3200 gas chromatograph/mass spectrometer system and is presently in the process of purchasing a data system to interface to it. The only shortcoming anticipated is the lack of personnel necessary to fully utilize the system.

- a. Deficiencies - None

5. Methodology

Copies of the officially published methods are available in the Unit. Documentation of how the methods are actually implemented in the laboratory is either not available or are in outline form. For some parameters (such as PCBs in oil), there is no documented method. All of the procedures employed were however appropriate.

- a. Deficiency - Some of the procedures used in the laboratory are not documented or poorly documented.

Recommendation - document all methods as they are used in the laboratory.

6. Quality Assurance

There is no documented quality assurance program in the Organic Unit. Quality control data is however, collected. There are no formalized or documented control limits; the individual chemist making the measurement uses his/her judgement to determine if the data is acceptable. The detection limits in some cases were not calculated from the data; some PNA data reviewed during the on-site inspection revealed that the detection limit reported was lower than what the data indicated.

General documentation of the specifics involved during sample analysis (weights, volumes, controls, final volume of the extracts) was vague especially for those samples which had been started during the previous physical years. (Especially, the fish samples). It was my conclusion, based on what I was told, that it would be virtually impossible to determine the quality of the data for these samples.

- a. Deficiency - There is no over all quality assurance program for the Organic Unit.

Recommendation - document the procedures to be used to insure and/or to generate data of known quality.

- b. Deficiency - There are no formalized or documented control limits.

Recommendation - Summarize all quality control results available and use it to generate acceptance/rejection criteria. Set up arbitrary limits for those parameters which data is not available.

- c. Deficiency - Detection limits are not calculated from the analytical results.

Recommendation - Define how the detection limit is to be calculated using the results (noise levels) of the measurements.

- d. Deficiency - Specific documentation regarding the sample preparation for those samples started one or two years ago is incomplete.

Recommendation - All those samples for which the documentation is nebulous should not be analyzed. Furthermore the recommended holding time for these sample extracts has expired.

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Appendix B

Bacteriology Section of Analytical Services Minnesota Department of Health

The Bacteriology Laboratory, Section of Analytical Services, Minnesota Department of Health, was certified January 19, 1979, to analyze water samples by the Multiple Fermentation Tube (MPN) and Membrane Filter (MF) methods for water regulated by the Safe Drinking Water Act (SDWA). Since many of the quality assurance requirements covered in the SDWA certification are identical to those required for analysis of 106 samples they will not be repeated at this time. The laboratory was reviewed for 106 methodology August 29 and 30, 1980. At that time various recommendations were made concerning laboratory operations. This report will deal with those recommendations and their resolution plus any new problems which may have arisen since then.

I. Organization

Recommendation (1979) - Implement the use of permanent technicians in the Bacteriology laboratory as rapidly as possible.

Resolution - A permanent part time employee has been hired. His schedule specifically includes weekend work. The agency is to be congratulated for this action.

II. Physical Facilities

The laboratory area is too small for the amount of work and the number of technicians who are on duty at peak work times, and has insufficient storage space. Also, there is very limited refrigerator space. The laboratory contains 460 ft². The recommended laboratory space for SDWA work is 200 ft² floor space and six linear feet of bench space per analyst. Because of efficient placement of equipment, the bench space is adequate but the floor space is extremely inadequate. Since the same laboratory is used for SDWA and NPDES work this space problem will be addressed as a deficiency during the re-certification evaluation in 1981-82. Also, Mr. Peacock is engaged in a limited study of Compylobacter and Giarda in drinking water. Since both these organisms are pathogens the work area where this work is being done should be segregated, to some extent, from the rest of the laboratory activities. This is not possible with the present space. Mr. Peacock should be commended for his interest in this area and should receive every encouragement since the number of water laboratories that engage this type of work is very limited and the organisms in question are important in outbreaks of water borne infections.

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Recommendations - Provide extra storage space and refrigeration at a location convenient to the laboratory. Also, investigate the feasibility of expanding the present laboratory.

III. Laboratory Equipment

At the time of the on-site review both 44.5°C water baths were defective. The circulation device on the large water bath was malfunctioning and the incubation temperature for the small water bath was erratic, indicating possibly a faulty thermostat.

Recommendation - The water baths must either be repaired or new ones purchased.

IV. General Practices

Recommendations (1979) - To conform to holding time requirements for fecal coliform samples.

- a. The laboratory should investigate the use of local laboratories which are approved for fecal coliform testing or other alternative procedures, such as field laboratory facilities.
- b. The sample containers should contain a special bottle of water for temperature determination.
- c. The laboratory should reject samples which do not provide date and time of collection.

Resolutions:

- a. The instructions for receipt of samples specifier six hours (copy attached).
- b. This has been done (see copy of Attachment 1).
- c. None have been received since this recommendation was made.

V. Specific Analytical Methods

Recommendation (1979) - Follow the official test procedure as specified. The laboratory may, if it wishes, submit documentation requesting an alternate test procedure. Another possibility which will save time is to use the membrane filter procedure for fecal coliform analyses.

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Resolution - The laboratory is presently following the official procedures and is collecting data to either substantiate or reject the necessity of transferring 48 hour presumptions in the EC (see Attachment 2).

VI. Quality Assurance

No comments

No recommendations

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Appendix C

Water and Air Chemistry Section of Analytical Services Minnesota Department of Health

I. Personnel

Three personnel deficiencies were noted in the Section of Analytical Services during August 19 and 20, 1980. No recommendations are made as to how the vacancies are to be filled since this is the prerogative of the Minnesota Health Department. The lack of personnel noted below, must be corrected if the Minnesota Health Department is to be responsive to the needs of the MPCA.

- a. Deficiency - Laboratory Director position is still vacant.
- b. Deficiency - Due to personnel freezes and normal personnel attrition, the Section of Analytical Services is understaffed in the above two Units. It is understaffed to the extent that the SAS cannot effectively respond to MPCA needs and cannot meet sample turn-around times. Personnel deficiencies during August 1980, were more severe than during August 1979.
- c. Deficiency - The QAO report of March 10, 1980 indicated that inadequate personnel were available for Auto Analyzer analyses and for quality assurance. It is apparent that adequate resources are being devoted to quality assurance; however, during August 1980, inadequate personnel were still inadequate in number for effective Auto Analyzer analyses. Personnel had been hired during 1979-80 for this type of analytical work, but were terminating their own employment for a variety of reasons. They were not being replaced as of August 1980. The Auto Analyzer personnel deficiencies cited in August 1979, therefore, were present August 20, 1980.

II. Methodology

During August 1979, the Air and Water Chemistry Unit, SAS was performing a certain number of nitrogen inhibited, long-term BOD measurements for the MPCA. Within Region V, long-term BOD measurements are becoming more popular for wasteload allocation studies or modeling purposes however, no reference method or reference technique exists for this parameter or parameters. Also, techniques commonly used for long-term BOD's must differ from the "Standard Methods" test for BOD₅.

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The SAS was performing carbonaceous nitrogen inhibited long-term BOD tests. The following deficiencies were noted in the SAS test. These deficiencies are not unique to Minnesota and have been observed throughout Region V.

- a. Deficiency - The SAS was not involved in MPCA's, planning of BOD study soon enough. For example, the SAS was not aware of BOD allocation study soon enough for sufficient BOD test bottles to be available. The SAS is not aware or familiar how resulting data are to be used so that proper test method techniques will be used. It is probable that the MPCA is unaware of the SAS's exact BOD test procedure and how certain variables, effect the resulting test data.

Recommendations - Future wasteload allocation studies must use closer planning and exchange of information between the MPCA and the SAS.

- b. Deficiency - For diluted river and waste samples, no seeded blank is subtracted from the oxygen depletion of the diluted samples. While this is at variance with the "Standard Methods," five day test for BOD, the blank subtraction is widely used and necessary for long-term BOD measurements. Because no reference method exists for long-term BOD, this cannot be considered a regulatory deficiency.

Recommendation - Implement better planning and exchange of information between MPCA and the SAS.

3. Deficiencies Corrected Since August 1979

A. Analytical Methodology

The analytical methods used by the two SAS Units evaluated in August 1980 are unchanged since August 1979.

1. The SAS's ammonia test procedure will soon be approved as an alternate test procedure for NPDES monitoring. Additional information has been requested from SAS to justify low-level ammonia measurements in surface waters in the presence of possibly interfering organic nitrogen compounds.

B. Quality Control

A visible, ongoing effort is in effect to implement a quality assurance program for the SAS. It is not yet in effect. The initial drafts of the SAS's quality assurance program is currently being reviewed by the QA0.

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The Metals Unit have continued their excellent quality control effort of 1979.

The Auto Analyzer group, for their associated analyses, have markedly improved the precision and accuracy of their nutrient data since 1979. Day-to-day evaluation of duplicate and "A + B controls" quality control audits have improved further since August 1979 and should be considered quite acceptable. The Water and Air Chemistry Unit has successfully implemented the quarterly analysis of reference samples.

The out-of-control situation (10% error) cited for mercury analyses in the March 10, 1979 report has been corrected by more careful preparation of mercury control solutions.

IV. Deficiencies Not Corrected Since August 1979

A. Analytical Methodology

The Kjeldahl nitrogen, total phosphorus, and phenolics test procedures used by the SAS are currently not approved for NPDES compliance monitoring. It is believed that future amendments to 40 CFR 136 will approve a block digester Kjeldahl nitrogen methodology, so that this deficiency may be eliminated.

The automated phenolics test procedure of the SAS has a certain utility for surface water monitoring, but has not been demonstrated to be equivalent to the reference method for NPDES monitoring. The same is true for total phosphorus. The block-digester method for total phosphorus should not be considered acceptable for low level phosphorus monitoring below 20 to 30 ug/l.

B. Quality Control

None

V. New Deficiency Not Identified During August 1979

During the August 19, 1980, evaluation visit to the Air and Water Chemistry Unit, the Unit's BOD methodology was evaluated in detail. The Unit needs to re-evaluate its BOD test procedure and markedly improve it. The test procedure must be upgraded so that unseeded dilution water has acceptable blank values, so that serial dilutions of a wastewater do not provide markedly different BOD test values, and so that control solution BOD test values do not differ by more than 10 to 15% and are not significantly biased from acceptable values. The Unit should investigate or re-evaluate:

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- a. Calibration procedures of their dissolved oxygen meter.
- b. Preparation and aging of dilution water so that unseeded blanks provide acceptable BOD₅ test values.
- c. Addition of nutrients to dilution water no more than 24 hours before the test is initiated.
- d. Sample seeding techniques for more consistent results.
- e. Preparation of consistent control solution (Glucose/Glutamic Acid).

Recommendation - Prior to any major work on their BOD test, SAS staff should visit at least two other State laboratories of Region V, having successful BOD testing procedure, to observe how other people perform the test, both for long term and five day BOD values.

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RECEIPT OF FECAL COLIFORM AND B.O.D. SAMPLES

1. Effective June 1, 1980, all samples requesting the fecal coliform or B.O.D. analysis must be shipped under refrigerated conditions. Samples should be refrigerated promptly after collection. Shipping conditions will be checked at the time of receipt into the laboratory.
2. A 120 ml plastic bottle with a black cap will be placed in each cooler permanently. This bottle has been filled with an ethyl alcohol solution (windshield washer fluid) to prevent freezing and to help identify it as a temperature control.
3. These bottles should be picked up at the Analytical Services receiving desk after May 19 and before May 30. Requests for temperature control bottles after May 30 will be processed through the regular bottle ordering procedure. Only one bottle per cooler is required.
4. When samples are received in the laboratory, the temperature should be taken immediately after the initial opening of the sample cooler. The temperature is taken by placing a thermometer in the control bottle without removing the bottle from the cooler. Close the cooler for two minutes; open, read and record the temperature to the nearest 0.5°C.

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5. Office personnel shall have the primary responsibility to measure and record the temperature.
6. When office personnel are not present to receive the sample, field personnel will be required to measure and record the temperature at the time of delivery to the laboratory.
7. Temperatures for each sample will be recorded to the nearest 0.5°C by office or field personnel on the data sheet in the column labeled "temperature", immediately to the right of the recorded field temperature. The two temperatures should be separated by a slash (see example).
8. Sample bottles should not be removed from the cooler until the temperature has been taken.
9. Office personnel will not accept any sample requesting the fecal coliform or B.O.D. analysis that has a temperature greater than or equal to 10°C at the time of receipt, unless the sample was collected within one hour of receipt. Any sample which is entirely or partially frozen will be accepted with an appropriate notation made on the data sheet.
10. Office personnel will reject the sample if the following information is not recorded on the data sheet:
 - 1) date of collection
 - 2) time of collection
 - 3) temperature upon receipt

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11. At no time shall a thermometer be placed directly into the sample itself.

12. Samples for fecal coliform or B.O.D. analysis submitted to the laboratory that require "Chain of Custody" procedures will be rejected if not received within 6 hours of collection and at a temperature of less than 10°C.

13. The next revision of data sheets should include places for: 1) time received by lab, and 2) temperature upon receipt (lab).

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Samples Collected By _____ Report To _____

Field Number	River, Town, Etc.	Sampling Point and Source of Sample
a		
b		
c		
d		
e		

This line for Lab use only		a	b	c	d	e
Sample Number						
Date Collected						
Time Collected						
Date Received by Lab						
Temperature, °C		10.0/5.5	10.0/5.5			
Dissolved Oxygen						
pH value, SL		013				
Total Residual Chlorine						
Fecal Coliform, MPN/100 ml		305				
Fecal Strep., No/100 ml		213				
Total Solids		001				
Suspended Solids		003				
Turbidity, NTU		011				
Calcium as CaCO ₃		025				
Magnesium as CaCO ₃		024				
Total Hardness as CaCO ₃		021				
Chloride as Cl		023				
5-Day B.O.D.		096				
Nitrification inhibited BOD ₅		053				
Total Phosphorus as P		059				
Orthophosphorus as P		063				
Organic Nitrogen as N		065				
Ammonia Nitrogen as N		064				
Nitrite + Nitrate Nitro		069				
Nitrite Nitrogen as N		067				
METALS (µg/l)	Arsenic as As	106				
	Cadmium as Cd	122				
	Total Chromium as Cr	129				
	Hexavalent Chromium	034				
	Copper as Cu	143				
	Iron as Fe	150				
	Lead as Pb	157				
	Manganese as Mn	164				
	Mercury as Hg	200				
	Nickel as Ni	171				
	Zinc as Zn	192				
Cyanide		066				
Phenol µg/l		085				
Oil and Grease		069				
Chlorophyll a, µg/l		450				
Kjeldahl Nitrogen		068				
*Flame/Furnace						

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Results are in mg/l except as otherwise noted

ANALYTICAL DATA

Report To

This line for Lab. use only.	A	B	C	D	E
Sample Number _____					
Date Collected _____					
Time Collected _____					
Temperature °F FIELD LAB 80 / 76 97 / 86					
Data Received by Lab _____					
Coliform { M.P.N per 100 -1. 302 group } Cor. = Comp. = organisms M.F.C. per 100 -1 306					
Total Solids ----- 001					
Turbidity ----- 011					
Color ----- 012					
Total Alkalinity as CaCO ₃ ----- 021					
Milkalkalinity as CaCO ₃ ----- C22					
pH value ----- 013					
Iron ----- 032					
Manganese ----- 033					
Chloride ----- 023					
Residual Chlorine -----					
Sulfate ----- 028					
Fluoride ----- 029					
Total Phosphorus ----- 059					
Nitrite Nitrogen ----- 067					
Nitrate Nitrogen+Nitrite ----- 066					
AMBS ----- 035					
Calcium as CaCO ₃ ----- 025					
Sodium ----- 026					
Potassium ----- 027					
Spec. Cond. micromhos/cm @ 25°C ----- 014					
pH at 50°F -----					
Formalindehyde ----- 055					
+-----					

% Bal. -----					

• התאחדות העובדים היא ארגון העובדים הראשון בישראל.

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Attachment 2

SUMMARY OFFECAL COLIFORM MPN STUDY

Following the E.P.A. survey of August 1979, the microbiology unit began following the Standard Methods requirement of transferring 48 hour presumptive gas positives to EC broth for all NPDES samples requesting the fecal coliform analysis. In addition, all samples requiring duplicate analysis for Quality Assurance (1 in 10) were also run according to Standard Methods. (Most of these QA samples were routine stream samples.)

Prior to the survey, data had indicated that transferring 48 hour presumptive positive tubes to EC for surface water samples had very little effect on sample results. To determine whether Standard Methods procedure could be modified for NPDES samples, the number of positive and negative tubes were recorded and the results tabulated.

A total of 113 surface water samples and 10 drinking water samples were examined by the Standard Methods MPN procedure from December 10, 1979 to June 25, 1980. The breakdown of samples by budget code and type is listed in table I.

Table II summarizes the EC positive samples from 48 hour positive presumptives. None of the 10 drinking water samples were positive in EC from the 48 hour transfers and are not included in the remaining tabulation.

Table III shows the MPN results from both procedures for the six positive samples.

A breakdown of the number of positive tubes in each phase of the procedure for both chlorinated and non-chlorinated samples is given in table IV.

A similar breakdown for NPDES and non-NPDES samples is presented in table V.

TABLE I

CODE	TYPE	CHLORINATED?			TOTAL
		YES	NO	UNKNOWN	
37,000	Drinking Water	0	10	0	10
127,000	Routine (x,y)	0	32	0	32
130,000	NWQSS (x,y)	0	22	0	22
121,000	NPDES	14	1	0	15
134,000	NPDES	1	9	0	10
141,000	NPDES	0	2	0	2
161,000	NPDES	2	27	3	32

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TABLE II

NUMBER OF EC POSITIVE SAMPLES FROM 48 HR. POSITIVE PRESUMPTIVES

CODE	<u>CHLORINATED</u>		<u>NON-CHLORINATED</u>		<u>UNKNOWN</u>		<u>TOTAL</u>	
	No.	%	No.	%	No.	%	No.	%
37,000	----	%	0/10	0%	----	%	0/10	0%
127,000	----	----	0/32	0	----	----	0/32	0
130,000	----	----	2/22	9	----	----	2/22	9
OTHER SUB- TOTAL	----	----	2/54	3.7	----	----	2/54	3.7
121,000	2/14	14.3	0/1	0	----		2/15	13.3
134,000	0/1	0	0/9	0	----		0/10	0
141,000	----		0/2	0	----		0/2	0
161,000	0/2	0	1/27	3.7	1/3	33.3	2/32	6.3
NPDES SUB- TOTAL	2/17	11.8	1/39	2.6	1/3	33.3	4/59	6.8
*TOTAL	2/17	11.8	3/93	3.2	1/3	33.3	6/113	5.3

* Excludes Drinking Water Samples

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TABLE III

SAMPLE NUMBER	CL.	24 HR. MPN	95% CONFIDENCE LIMITS	24 + 48 HR. MPN	WITHIN 95% CONFIDENCE LIMITS
			24 HR. MPN		
121467	YES	3300	1100-9300	7900	YES YES
121495	YES	50	< 5-130	80	YES YES
130247	NO	< 20*	-----	20	--- YES
130270	NO	4600	1600-12,000	6300**	YES YES
161398	NO	< 20*	-----	20	--- YES
161458	UNKN.	< 20*	-----	80	--- YES

* NO CONFIDENCE LIMITS GIVEN

** IMPROBABLE CODE

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TABLE IV

	CHLORINATED		NON-CHLORINATED	
	24 HR. GROUP	48 HR. GROUP	24 HR. GROUP	48 HR. GROUP
PRESUMPTIVE POSITIVE	32.9 168/510	5.8 .20/342	35.3 755/2140	15.2 210/1385
EC POSITIVE	72.0 121/168	15.0 3/20	67.5 510/755	1.4 3/210

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TABLE V

	NPDES		NON-NPDES	
	24 HR. GROUP	48 HR. GROUP	24 HR. GROUP	48 HR. GROUP
PRESUMPTIVE POSITIVES	31.8 531/1670	9.3 106/1139	38.3 410/1070	20.3 134/660
EC POSITIVE	76.8 408/531	6.6 7/106	57.3 235/410	1.5 2/134

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OBSERVATIONS AND CONCLUSIONS

1. Overall, 5.3% of the 113 surface water samples were positive in EC when transferred from 48 hour presumptive positive tubes.
2. 68.3% $\left(\frac{643}{941}\right)$ of the 24 hour Lauryl Tryptose positives were positive in EC while only 3.8% $\left(\frac{9}{240}\right)$ of the 48 hour Lauryl Tryptose positives were positive in EC.
3. To determine whether the differences observed in tables II, IV and V are significant, several Chi Square Tests were performed with the following results:
 - 1) Based on positive sample results at 48 hours, there is no significant difference between chlorinated and non-chlorinated samples.
 - 2) Based on positive sample results at 48 hours, there is no significant difference between NPDES and non-NPDES samples.
 - 3) Based on 48 hour EC tube results, there is a significant difference between chlorinated and non-chlorinated sample results.
 - 4) Based on 48 hour EC tube results, there is a significant difference between NPDES and non-NPDES samples.
4. For the six samples in which a positive EC result occurred from transferring 48 hour positive presumptive tubes, the larger MPN value which resulted was within the 95% confidence limits of the 24 hour value and conversely.
5. The results of the present study are in close agreement with the 1975 study.
6. The extra time and media spent on the Standard Methods procedure is not substantial and the 24 hour delay in reporting not critical.
7. Because of the few numbers of positives and the conflicting conclusions of the Chi Square Test, additional data should be collected.

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